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Picoindentation Hardness Measurements Using Atomic Force Microscopy

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Abstract

An atomic force microscope (AFM), with a specially prepared diamond tip, has been modified to measure indentation hardness with an *indentation depth as low as 1 nm*. This indentation depth is much smaller than the depth of more than 20 nm that have been reported to date. The AFM indentation technique allows the hardness measurements of surface monolayers and ultrathin films in multilayered structures at very shallow depths and low loads. The picoindentation hardness of single crystal silicon are measured using this technique. A subtraction technique is also described which allows the actual hardness measurements of rough surfaces such as magnetic thin film rigid disks.

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For hardness measurements of bulk materials and thin films, various indentation techniques are available. In order to get accurate measurements of hardness of films, the film thickness should be at least five times the depth of penetration¹. Pethica et al.² developed a depth sensing instrument in which small loads can be applied so that a minimum depth of penetration of about 20 nm can be achieved. As it becomes necessary to measure hardness of ultra thin films (10 nm or less) such as in computer industry³ and microsystems⁴, new techniques are needed to make measurements at very shallow depth. In this paper we report the hardness measurement studies carried out at indentation depth as low as 1 nm using an atomic force microscope (AFM).

We have modified commercial AFM (Nanoscope III from Digital Instruments, Inc., Santa Barbara, CA) and used a diamond tip mounted on a stiff cantilever beam, to measure indentation hardness. The tips were made from a single-crystal natural diamond. These diamond tips were ground to the shape of a three-sided pyramid with an apex angle of 80° whose point is sharpened to a radius of about 100 nm. Scanning electron microscopy (SEM) micrograph of the three-sided pyramidal diamond tip is shown in Fig. 1. These tips were bonded with conductive epoxy to a gold-plated 304 stainless steel spring sheet that acts as a cantilever. Stiffness of the cantilever beam was changed by changing the length of cantilever beam. The normal load was determined by multiplying cantilever spring constant by the cantilever deflection. Picoindentation measurements can be made in the normal load range of 10 μN to 150 μN with a cantilever stiffness of 50 N/m. During indentation experiments, the scan size was set to zero, in order for the tip to continuously press the sample surface for about two seconds. Indentation marks were generated on the sample surface as a result of the normal load being applied by the tip. The surface was imaged before and immediately after the indentation at a normal load of about 0.5 μN . Picohardness was calculated by dividing the indentation load by the projected residual area. A subtraction technique is described which allows the actual hardness measurements of rough surfaces such as magnetic disk surfaces³. The Si(111) samples measured in this letter, were ultrasonically cleaned in methanol for 20 minutes and dried in dry nitrogen atmosphere prior to mounting on AFM. Picohardness measurements were carried out in the ambient atmosphere.

Figures 2(a) and (b) show the gray scale plots and line plots of inverted images of indents made on Si(111) at normal loads of 60, 65, 70 and 100 μN . Triangular indents can be clearly observed with very shallow depths. It is found that below a normal load of 60 μN indentation marks are unobservable. At a normal load of 60 μN indentation marks are observed and the depth of penetration is about 1 nm. As we increase the normal load, the indentation marks become clearer and indentation depth increases. Figure 3 shows a plot of hardness and normal load as a function of indentation depth. The depth of indentation increases with an increase in normal load. We note that hardness at a small indentation depth of 2.5 nm is 16.6 GPa and it drops to a value of 11.5 GPa at depth of 12 nm and normal load of 130 μN . This hardness data is comparable to the nanohardness data reported by Pharr et al.⁵. High hardness at shallow depth probably arises from the hard surface (oxide) films. If the silicon material is used at very light loads such as in micro systems, high hardness of surface films would protect the surface until it is worn.

For the case of hardness measurements made on magnetic thin film rigid disk at low loads, indentation depth is on the same order as the variation in the surface roughness. For accurate measurements of indentation size and depth, it is desirable to subtract the original (unindented) profile from the indented profile. We developed an algorithm for this purpose. Because of hysteresis, a translational shift in the sample plane occurs during the scanning period, resulting in a shift between images captured before and after indentation. Therefore, we need to shift the image for perfect overlap before subtraction can be performed⁶. To accomplish our objective, a small region on the original image was selected and the corresponding region in the indented image was found by maximizing the correlation between the two regions. (Profiles were plane-fitted before subtraction.) Once two regions were identified, overlapped areas between the two images were determined and the original image was shifted with the required translational shift and then subtracted from the indented image. An example of profiles before and after subtraction is shown in Fig. 4. It is easier to measure indent on the subtracted image. At a normal load of 140 μN hardness value of an unlubricated, as-polished magnetic thin film rigid disk (rms roughness = 3.3 nm) is 9.0 GPa and indentation depth is 40 nm.

In summary, the hardness measurement at an indentation depth of 1 nm were performed for the first time on the surface of the Si(111) sample. We have shown that an AFM indentation technique allows hardness measurements of monolayer and ultrathin film in multilayered structures.

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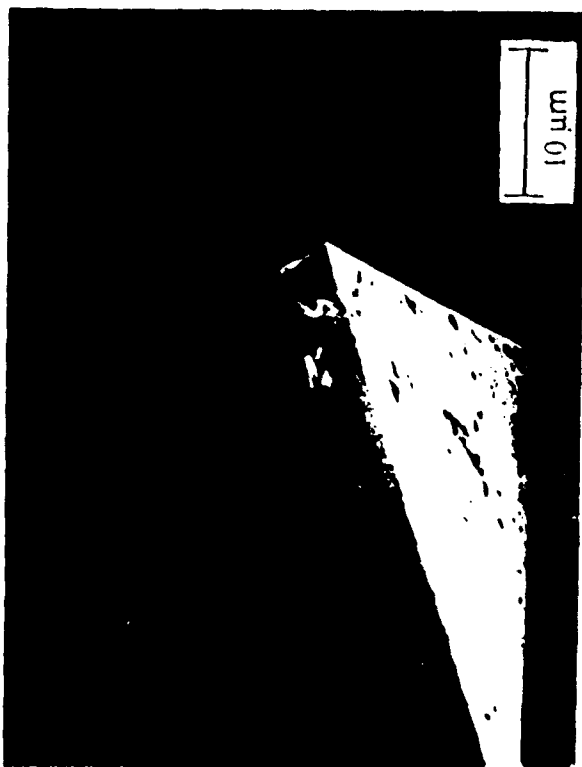
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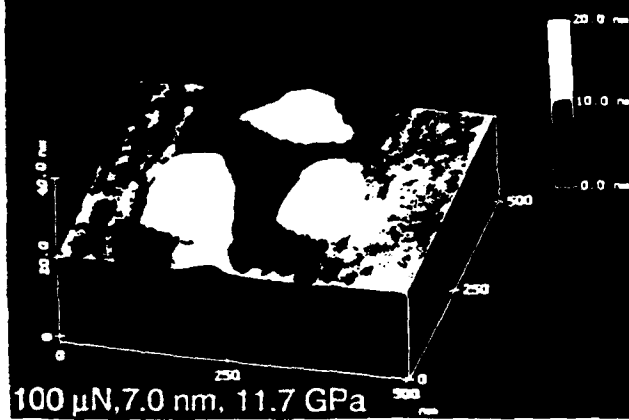
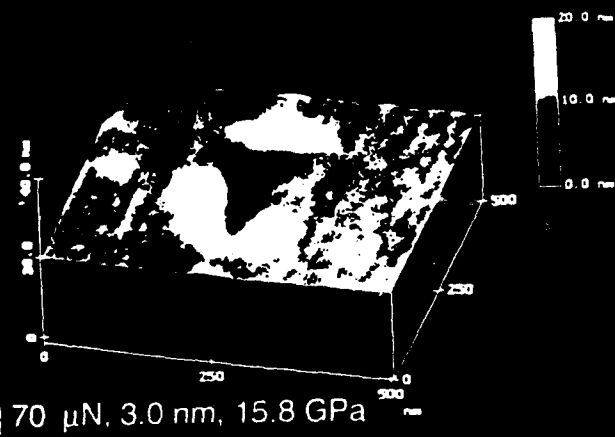
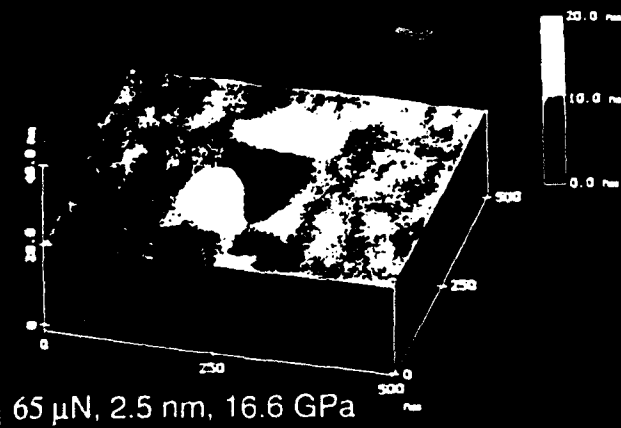
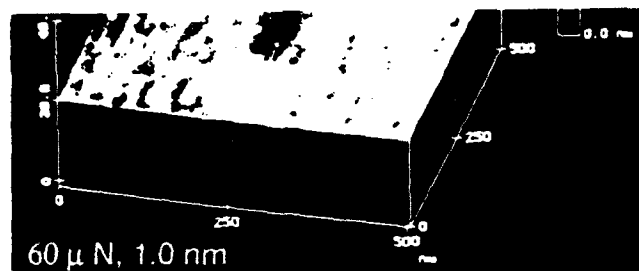
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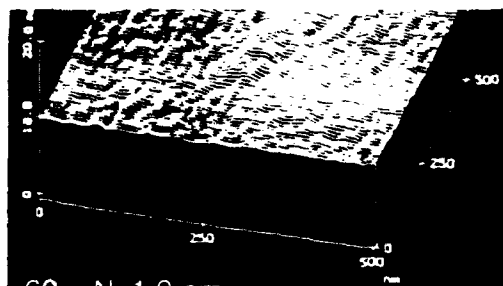
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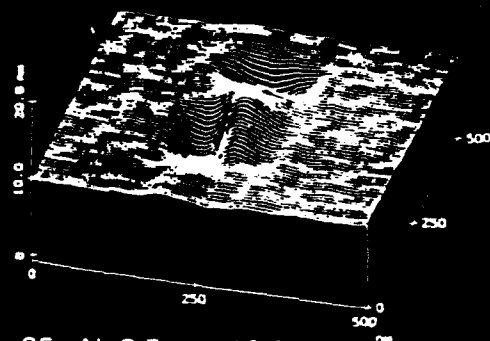
1. SEM micrograph of a three-sided pyramidal (natural) diamond tip.
2. (a) Gray scale plots and (b) line plots of inverted images of indentation marks on Si(111) at various loads. Loads, indentation depths and hardness values are listed in the figure.
3. Picohardness and normal load as a function of indentation depth for Si(111) sample.
4. Images with indentation marks generated on an unlubricated, as-polished magnetic thin film rigid disk at 140 μN (a) before subtraction, and (b) after subtraction.



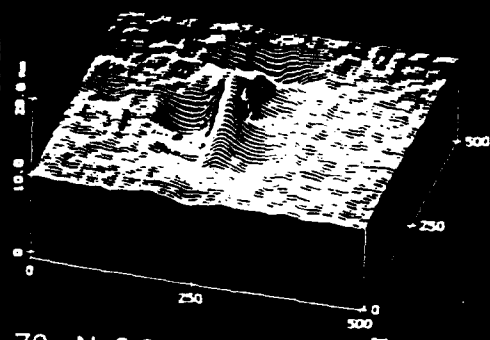




60 μ N, 1.0 nm



65 μ N, 2.5 nm, 16.6 GPa



70 μ N, 3.0 nm, 15.8 GPa

